DISTILLATION FOR ACID SOLUBLE AND ACID INSOLUBLE SULFIDES
EPA 9030 B Rev 2 12/1996

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Are all aqueous samples and effluents preserved with zinc acetate and NaOH (4 drops 2 N ZnAc per 100 mL of sample and pH > 9 with 6 N NaOH)?	6.2				
Are samples analyzed within 7 days?	6.2				
For solids is enough ZnAc added to fill the surface of the solid surface until moisten?	6.2				
Are samples stored at 4 C and headspace free?	6.2				
Is distillation unit set up per Figure 1 (24/40 connections reduced to ¼ inch tube).	4.1				
Are solids suspended in distillation unit so not to disrupt the stirring bar?	6.3.1				
For aqueous samples is sample shaken and transferred to graduate cylinder and then transferred to distillation unit weighing the cylinder full and after transfer?	6.3.2				
If the sample contains chucks, is it milled, weighted, and 250 mL of water added?	6.3.3				
If aqueous and large amount of solids, is the phases separated and proportionally weighed and water added to 150 mL?	6.3.4				
If sample contains solids that absorb water and swell, is sample limited to 25 gms?	6.3.5				
If sample can only broken, are they cut and if also contain liquid, is it proportional weighted?					
If samples contain hard objects, are they weighed and discarded and % reported and is used in the calculation?					
Notes/ Comments:					

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Acid Soluble Sulfide Was a preliminary analysis performed to determine the amount of H2SO4 to reduce pH to 1 (Choose sample size to give between 0.2 – 50 mg of S. Place know sample amount in beaker and dilute to 100 mL. How much H2SO4 to get pH = 1)?	7.1				
Is distillation unit in 70 C bath?	7.1.2.1				
Is 10 + /- 0.5 mL 0.5 M ZnAc, 5 mL + /- 0.1 mL of 37% formaldehyde, and 100 + /- water placed into gas scrubber bottle?	7.1.2.3				
Is sample added and if appropriate diluted to 200 mL	7.1.3				
Is N2 purged at 25mL/min (~5 bubbles/second)?	7.1.5				
Is sample heated to 70C and the amount of H2SO4 per 7.1 + 50 mL added per the funnel at a rate of 5 mL/min?	7.1.6				
Is the distillation maintained at 70C for 90 minutes (start to finish)?	7.1.7				
Acid Insoluble Sulfide If matrix is a dry solid, is sample weight determined to get 0.2 to 50 mL S, crushed to 1 mm particle size and 50 mL water added?	7.2.2				
If aqueous sample, is a max of 50 g of sample and no water is added (if less than 50 g need to get to 0.2 – 50 range, add water to 50 mL?	7.2.3				
If moist solid, is the water content must be measured and the water included in the 50 mL added?	7.2.4				
Is sample, 5 g SnCl2, and water per above added to distillation flask?	7.2.5				

Notes/ Comments:

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Is 100 + /- 2 mL ZnAc/NaAc buffer and 5 + /- 0.1 mL formaldehyse added to each scrubbing bottle?	7.2.6				
Is the N2 purge set up as 7.15, stirrer set as fast as possible, and is 100 mL + /- 1 mL of 9.8 N HCl added?	7.2.7 – 7.2.9				
Is the water bath at 100 C and distillation performed for 90 minutes?	7.2.10				
For both preparations is EPA 9034 or EPA 9215 used?	7.1.8 7.2.11				
Are LOD determined using 7 replicates at 0.45 4.5 mg/L.	9.3				
Notes/ Comments:					